Comparison of Calculated d-values with d-values on JCPDS Cards

A Thesis

Presented in Partial Fulfillment of the Requirements
for the Degree Bachelor of Science

by
Michael William Smith

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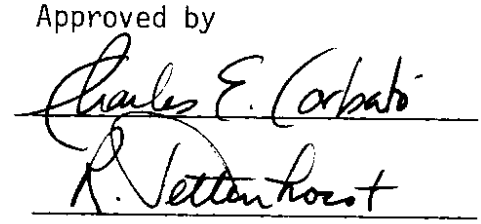
Approved by



Advisors
Department of Geology
and Mineralogy

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INTRODUCTION

The objective of this research was to investigate the X-ray data cards published by the Joint Committee on Powder Diffraction Standards (JCPDS) for errors in indexing, printed d-values, cell dimensions, or a combination of these. This was done by comparing d-values on the card with d-values calculated using equations which relate d-values to hkl and cell dimensions. Explaining the discrepancies is a more difficult problem because there are many things that can cause differences. For instance, cell parameters may be incorrect. Sometimes d-values are given Miller indices that do not coincide with those allowed in the space group of the mineral or are in error for the reflection under consideration. Another possibility is that the mineral had impurities in it that produced d-values that are uncharacteristic of the mineral being studied. Other possibilities that might produce errors are incorrect space groups or incorrect crystal systems. Among all these possibilities, though, there is one kind of inaccuracy that is prevalent; d-values on the cards differ from those calculated using the cell parameters for that mineral. These inaccuracies may be attributable to observational errors in reading 2-theta. This work is an attempt to find if any errors are present in a selected group of minerals, and to determine the cause, if possible.

PROCEDURE

Two computer programs, prepared by C. E. Corbato, were utilized. First, an hkl generator program was used to determine all possible Miller indices and their corresponding d-values from the given space group and cell parameters on the JCPDS card. A minimum d-value was put in the program to place a lower limit on the d-values generated. The particular space group of a

given mineral is determined by the Miller indices of sets of planes which are extinguished owing to the atomic arrangement. The program prints out the indices that are allowed for a given space group along with their corresponding d-values and orders the d-values by value from greatest to least. The d-values are calculated using the appropriate equation which depends on the crystal system of the mineral.

$$\text{Isometric:} \quad d_{hkl} = \frac{a_o}{\sqrt{h^2+k^2+l^2}} \tag{1}$$

$$\text{Tetragonal:} \quad d_{hkl} = \frac{1}{\sqrt{\frac{h^2+k^2}{a_o^2} + \frac{l^2}{c_o^2}}} \tag{2}$$

$$\text{Orthorhombic:} \quad d_{hkl} = \frac{1}{\sqrt{\frac{h^2}{a_o^2} + \frac{k^2}{b_o^2} + \frac{l^2}{c_o^2}}} \tag{3}$$

$$\text{Hexagonal:} \quad d_{hkl} = \frac{1}{\sqrt{\frac{4}{3}\frac{h^2+hk+k^2}{a_o^2} + \frac{l^2}{c_o^2}}} \tag{4}$$

These calculated Miller indices and d-values were then compared with those on the JCPDS card to determine if there are any great dissimilarities. If there were, then the second program was used.

The second program gives two tables. One table is titled Refinement Minimizing the Sum of the Squares of the Residuals of 1/D**2. The other is named REfinement Minimizing the Sum of the Squares of the Residuals of Theta. This program calculates cell dimensions for the mineral using previously

assigned Miller indices, a set of observed d-values or 2-theta values, and the mineral's crystal system as input. The program does this by using a linear least squares procedure. This procedure's premise, according to Cullity (1956, p. 335) is that "the most probable value of the measured quantity," 2-theta in our case, "is that which makes the sum of the squares of the errors a minimum." An example of one way this is done is given in Appendix 1 (prepared by C. E. Corbato). Also in the program is a value for the wavelength (λ). From this information the remainder of the first table of program one is calculated by means of the following relations:

$$2\text{-theta} = 2\arcsin^{-1}(\lambda/2d)$$

D(CALC) is calculated using equations 1, 2, 3, or 4 depending on the crystal system of the mineral.

Residuals

$$1/D^{**}2 = \frac{1}{D(OBS)^2} - \frac{1}{D(CALC)^2}$$

2-theta=observed(2-theta)- calculated(2-theta)

In the second part of program one the cell dimensions are computed in a non-linear least-squares procedure which gives slightly better results. In this program the sum of the square of the residuals of theta is minimized. The rest of the calculations are carried out in the same way as in the first portion of the program.

Overall, there are three different values for d; from the JCPDS card, from the hkl generator (from Program 1), and a calculated d-value (from Program 2). The d-values on the card and the d-value from the hkl generator should be very close. This so because they are associated by the cell dimensions. The hkl generator program used the cell dimensions on the card to obtain d-values. The d-values on the card were used by

other workers to calculate the cell dimensions given on the card. If there is disagreement between the two values then it may be due to an error in reading 2-theta from powder films or X-ray strip charts on the original pattern from which the card data were tabulated. These will be noted.

Next, JCPDS d-values and calculated d-values from the refinement program are compared. This is where differences begin to occur. When the calculated d-values are computed, new cell dimensions are used. If the difference between these cell dimensions and those on the JCPDS card are substantial then residuals produced by the refinement program will be great.

RESULTS

Embolite

14-255

d	2.81	1.99	1.26	3.24	Ag(Cl,Br)
I/I$_1$	100	60	40	20	Silver Chloride Bromide Embolite

Rad. CuKα λ 1.5418 Filter Ni Dia. 57.3mm
Cut off I/I$_1$
Ref. Berry and Thompson, Geol. Soc. Am. Mem. 85 215 (1962)

Sys. Cubic S.G. Fm3m (225)
a_o 5.626 b_o c_o A C
α β γ Z 4 Dx
Ref. Ibid.

ε nωβ εγ Sign
2V D 5.7-5.8 mp Color Colorless to gray
Ref.

Sample from Broken Hill, New South Wales, called Bromian Cerargyrite in Dana; most analyses show Cl>Br.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.26	20	111			
2.81	100	200			
1.989	60	220			
1.698	10	311			
1.623	30	222			
1.408	10	400			
1.290	5	331			
1.258	40	420			
1.152	30	422			
0.993	10	440			
.938	20	600,442			
.899	10	620			
.849	10	622			

Figure 1 JCPDS card for embolite

The difference between the calculated cell dimensions from the refinement and that on the card of this cubic mineral is only .001 Angstroms(A)(compare cell dimensions in Figure 1 with those of the refinement in Appendix 2.1). This shows up as some small differences between calculated and observed values of 1/D**2, of which the largest is .00437A^{-2}. This corresponds to an error in the reading of 2-theta of .299° 2-theta

for the (422) reflections(Appendix 2.1). The JCPDS data for embolite are considered to be essentially correct.

Hardystonite

12-453

d	2.87	3.09	3.71	5.02	$Ca_2ZnSi_2O_7$
I/I$_1$	100	60	50	35	Calcium Zinc Silicate — Hardystonite

Rad. CuK$_{\alpha_1}$ λ 1.5405 Filter Ni Dia.
Cut off 3%⊖ I/I$_1$ Diffractometer
Ref. L. G. Berry, Queen's University, Kingston, Ontario, Canada

Sys. Tetragonal S.G. $P\bar{4}2_1m$ (113)
a_o 7.823 b_o c_o 5.013 A C 0.641
α β γ Z 2 Dx 3.39
Ref. Ibid.

ε α 1.6568(Na) nωβ 1.6691(Na) εγ Sign −
2V D 3.443 mp Color Light brown to pinkish
Ref. Palache, U.S. Geol. Survey, Professs. Papers 180, 94 (1935)

Sample from Franklin, New Jersey, USA.
Analysis (Palache) %: 36.59 SiO_2, 0.77 Al_2O_3, 0.42 Fe_2O_3, 22.47 ZnO, 1.23 MnO, 35.16 CaO, 1.47 MgO, 1.99 P_2O_5.
Melilite group.

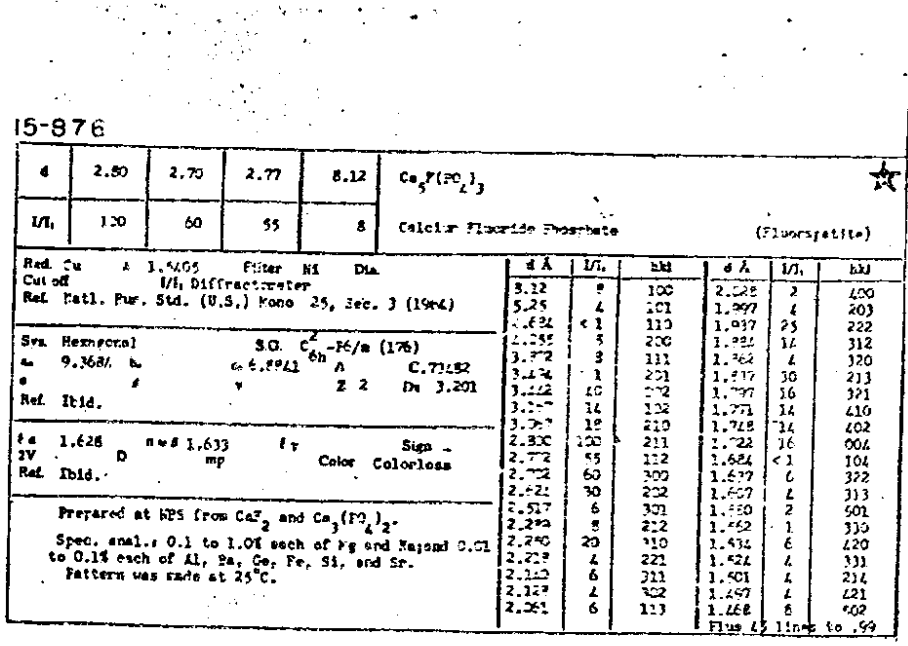
d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
5.018	35	001	1.761	35	312
4.220	10	101	1.730	8	331
3.711	50	211	1.64	40	103,322
3.493	6	210	1.600	8	113
3.085	60	203	1.537	8	203
2.951	6	ni	1.508	10	213
2.868	100	211	1.468	6	511
2.779	12	220	1.431	8	223
2.506	16	002	1.407	4	303
2.473	30	310	1.395	6	521
2.420	12	221	1.385	6	313
2.384	14	102	1.33	40	502,432
2.314	8	301	1.305	4	512
2.283	10	112	1.303	4	600
2.219	6	311	1.256	8	004,413+
2.037	18	212	1.239	6	620,104+
1.991	2	321			
1.949	2	400			
1.896	6	410			
1.858	12	222			

Figure 2 JCPDS card for hardystonite

The diffenence between cell demensions from the refinement and those on the card of this tetragonal mineral is .0034A for a_o and .003A for c_o (compare cell dimensions in Figure 2 with those of the refinement in Appendix 3.1). Some variation in the residuals of 2-theta can be found too, the greatest of which is .209° 2-theta for the (512) reflection(Appendix 3.1). Also, the 1.239A d-value on the card is indexed as either (620) or (104). According to the hkl generator this reflection should be indexed as (333)(Appendix 3.2). The unindexed d-value, 2.951A(Figure 2) is probably an impurity in the mineral since the hkl generator program produced no d-value close to 2.951A for the given cell dimensions and crystal system. Therefore, the JCPDS card data appear to contain one error in the assignment of the Miller indices for the d-value of 1.239A.

Fluorapatite

The difference between a_o from the card and the a_o from the refinement program is .0028A. The difference in c_o is .0007A(compare cell dim-



15-876

d	2.80	2.70	2.77	8.12	$Ca_5F(PO_4)_3$
I/I1	100	60	55	8	Calcium Fluoride Phosphate (Fluorapatite)

Rad. Cu λ 1.5405 Filter Ni Dia.
Cut off I/I1 Diffractometer
Ref. Natl. Bur. Std. (U.S.) Mono 25, Sec. 3 (1964)

Sys. Hexagonal S.G. C_{6h}^2-P6/m (176)
a_o 9.368A b_o c_o 6.8941 A C.7362
α β γ Z 2 Dx 3.201
Ref. Ibid.

εα 1.628 nωβ 1.633 εγ Sign –
2V D mp Color Colorless
Ref. Ibid.

Prepared at NBS from CaF_2 and $Ca_3(PO_4)_2$.
Spec. anal.: 0.1 to 1.0% each of Mg and Na and 0.01 to 0.1% each of Al, Ba, Ce, Fe, Si, and Sr.
Pattern was made at 25°C.

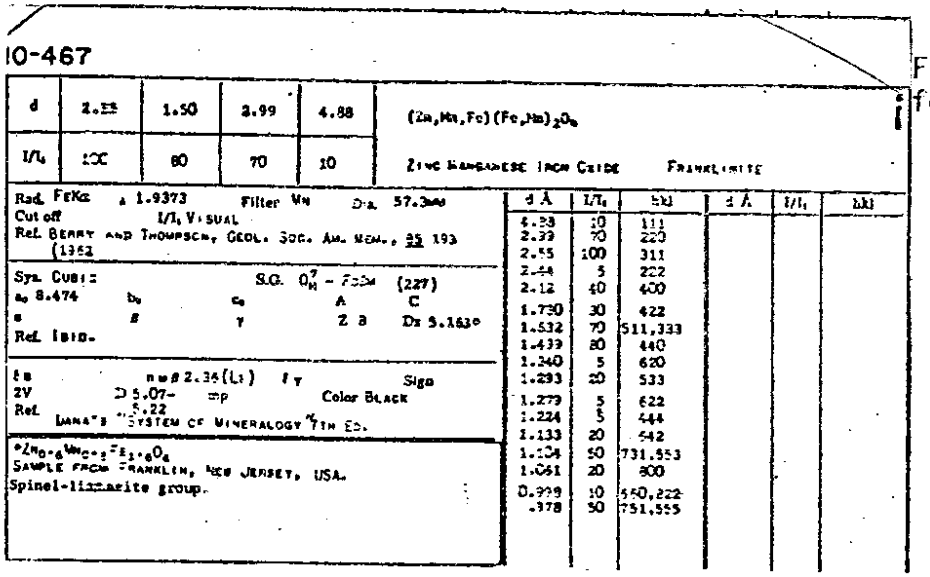
d Å	I/I1	hkl	d Å	I/I1	hkl
8.12	8	100	2.028	2	430
5.25	4	101	1.997	4	203
4.684	< 1	110	1.937	25	222
4.055	5	200	1.886	14	312
3.972	8	111	1.863	4	320
3.674	1	201	1.817	30	213
3.442	40	002	1.797	16	321
3.167	14	102	1.771	14	410
3.067	18	210	1.748	14	402
2.800	100	211	1.722	16	004
2.772	55	112	1.684	< 1	104
2.702	60	300	1.639	6	322
2.621	30	202	1.607	4	313
2.517	6	301	1.550	2	501
2.293	8	212	1.462	1	330
2.240	20	310	1.434	6	420
2.219	4	221	1.424	4	331
2.113	6	311	1.401	4	214
2.029	4	302	1.397	4	421
2.001	6	113	1.368	6	402

Plus 45 lines to .99

Figure 3 JCPDS card for fluorapatite

ensions in Figure 3 with those of the refinement in Appendix 4.1). These are small differences. Accordingly, the data taken from the card shows the greatest difference in 2-theta is .041° for the (300) reflections(Appendix 4.1). The JCPDS card data for fluorapatite appear to be exceptionally good.

Franklinite



10-467

d	2.53	1.50	2.99	4.88	$(Zn,Mn,Fe)(Fe,Mn)_2O_4$
I/I1	100	80	70	10	Zinc Manganese Iron Oxide Franklinite

Rad. FeKα λ 1.9373 Filter Mn Dia. 57.3mm
Cut off I/I1 Visual
Ref. Berry and Thompson, Geol. Soc. Am. Mem., 85 193 (1962)

Sys. Cubic S.G. O_h^7 - Fd3m (227)
a_o 8.474 b_o c_o A C
α β γ Z 8 Dx 5.163
Ref. Ibid.

εα nωβ 2.36(Li) εγ Sign
2V D 5.07- mp Color Black
Ref. 5.22
Dana's System of Mineralogy 7th Ed.

$(Zn_{0.6}Mn_{0.4})(Fe_{1.6}Mn_{0.4})O_4$
Sample from Franklin, New Jersey, USA.
Spinel-limnanite group.

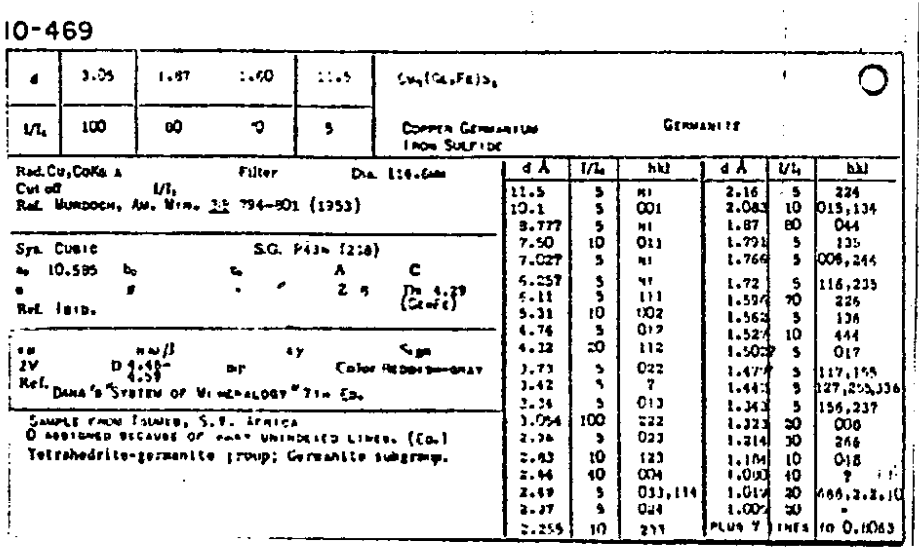
d Å	I/I1	hkl	d Å	I/I1	hkl
4.88	10	111			
2.99	70	220			
2.55	100	311			
2.44	5	222			
2.12	40	400			
1.730	30	422			
1.632	70	511,333			
1.499	80	440			
1.340	5	620			
1.293	20	533			
1.279	5	622			
1.224	5	444			
1.133	20	642			
1.104	50	731,553			
1.061	20	800			
0.999	10	660,822			
.978	50	751,555			

Figure 4 JCPDS card for franklinite

Franklinite shows a difference of .0028A between a_o on the card and a_o from the refinement(Appendix 5.1). Only minor discrepancies are observed for residuals of 2-theta, of which the largest is .168° for the

(800) reflection (Appendix 5.1). The JCPDS card data for franklinite are apparently correct.

Germanite



10-469

d	3.05	1.87	1.60	1.45
I/I1	100	80	70	5

Cu2(Ga,Fe)S4

Copper Germanium Sulfide — Germanite

Rad. Cu,CoKα λ — Filter — Dia. 114.6mm
Cut off — I/I1
Ref. Murdoch, Am. Min. 38 794-801 (1953)

Sys. Cubic — S.G. P43m (218)
a0 10.585 — b0 — c0 — A — C
α — β — γ — Z 2 — Dm 4.29 (Calc)
Ref. Ibid.

εα — nωβ — εγ — Sign
2V — D 4.48 4.59 — mp — Color Reddish-gray
Ref. Dana's System of Mineralogy 7th Ed.

Sample from Tsumeb, S.W. Africa
O assigned because of many unindexed lines. (Co)
Tetrahedrite-germanite group; Germanite subgroup.

d Å	I/I1	hkl	d Å	I/I1	hkl
11.5	5	NI	2.16	5	224
10.1	5	001	2.083	10	015,134
8.777	5	NI	1.87	80	044
7.50	10	011	1.791	5	135
7.027	5	NI	1.766	5	008,244
6.257	5	NI	1.72	5	116,235
6.11	5	111	1.596	70	226
5.31	10	002	1.562	5	136
4.76	5	012	1.524	10	444
4.32	20	112	1.502	5	017
3.73	5	022	1.477	5	117,155
3.42	5	?	1.443	5	127,255,336
3.34	5	013	1.343	5	156,237
3.054	100	222	1.323	30	008
2.96	5	023	1.314	30	266
2.83	10	123	1.184	10	018
2.64	40	004	1.093	40	?
2.49	5	033,114	1.014	30	466,2.2.10
2.37	5	024	1.002	30	-
2.255	10	211	PLUS 7	LINES	to 0.1063

Figure 5 JCPDS card for germanite

Comparing the hkl generator d-values(Appendix 6.1) with those on the JCPDS card many dissimilarities can be seen. First of all, many d-values appear on the card that do not appear on the hkl generator output. The first three values on the card are not allowable within space group number 218($P\bar{4}3m$), with a cell dimension of 10.585A. The second value among these three is given Miller indices which are not allowed in this space group. These Miller indices, (001), along with their odd orders of reflections should be cancelled out because of interleaving of other planes in this space group. The first d-value that does correspond to one on the hkl generator output is 7.50A. The next three have no matches on the hkl generator output. One of these with the value 6.11A is indexed as (111) but this reflection is not allowed in this space group for the same reason as above. Another d-value that is not on the hkl generator output is 3.42A. Galena, which is a mineral intimately associated with germanite according to Palache(1944, p. 386), has 3.429A as one of its d-values. It is possible that galena is responsible for this peak. The

d-value, 2.96A, when applied to the equation $a_o^2/d^2=h^2+k^2+l^2$, gives 12.79 for $h^2+k^2+l^2$. This cannot be indexed as (023), as it is on the card. This d-value may be due to some other mineral. Both galena and tennantite have d-values close to 2.96A, 2.969A and 2.94 respectively. Both of these minerals are intimately associated with germanite and either galena or tennantite may be responsible for this d-value. Calculating for $h^2+k^2+l^2$ with a d-value of 1.502A, 49.66 is obtained. This cannot be reasonably indexed as (017) as it is on the card. The d-value of 1.479A is indexed incorrectly. (117) and (155) are not allowed in this space group. Another problem with the d-values on this card seems to be an oversight. The d-value of 1.080 is unindexed. The value for $h^2+k^2+l^2$ for this d-value is 96.06. This can be indexed with indices (844).

For the above reasons it was decided to investigate germanite in more detail. Murdoch(1953, pp. 794-801) did the original work from which the card data were taken. He concluded that germanite is psuedo-isometric and shows some deviations from a truly isometric pattern. These deviations may account for the unindexed d-values and the values with question marks beside them on the JCPDS card.

An X-ray pattern of germanite was run. The sample used was from the type locality for germanite in Tsumeb, South-West Africa. Murdoch's material was from the same location.

The germanite was powdered to a grain size of less than 62 microns. The Phillips goniometer was run at a rate of ½ degree 2-theta per minute. The power was set at 35kV and 10ma. A Geiger detector was used. The radiation was copper with a nickel filter.

Before comparing d-values from the X-ray pattern with d-values from the hkl generator program, the d-values of germanite were calibrated with the d-values of quartz which was used as an external standard. This was

done by taking accurately known values of 2-theta, given by Frondel(1962, pp. 26-33) for quartz and comparing them with 2-theta values from an X-ray strip chart of quartz. The differences were then calculated and plotted on a graph against the value of 2-theta. A straight line was then drawn on the graph to follow the trend of the points. A reproduction of this graph is shown in Appendix 6.2. It was noted that the 2-theta values from the X-ray pattern of quartz were always less than the true(Frondel) values of 2-theta. This difference decreased with increasing 2-theta. Germanite was calibrated from this graph as follows: if a peak at 2-theta of 21.6° was observed the calibration shows that .04° 2-theta should be added to this. The corrected 2-theta is 21.64° 2-theta. For more information on this calibration method see Cullity(1956, p. 342).

As can be seen from the refinement based on corrected 2-theta values from the X-ray strip chart(Appendix 6.3) many of the values on the JCPDS card do not appear. Also the difference between calculated and observed 2-theta values is, in some cases, very large. In one case, the (10.7.3) planes have a difference of over 1° 2-theta(Appendix 6.4). If one assumes that the calibrated 2-theta values are correct, then either the crystal system or space group of germanite is incorrect. Since single crystals of germanite have never been observed it is difficult to determine the crystal system. Also, apparently, there is no agreement as to what is the true space group of the mineral. Palache(1944, p. 385) picks the space group as $F\bar{4}3m$. Murdoch(1953, p. 796) believes the space group of germanite because he considered it to be isostructural with colusite and renierite and crystals of these two minerals have been observed.

The errors on the JCPDS card for germanite may represent some problems as to what space group and crystal system the mineral belongs to.

CONCLUSIONS

Overall, the JCPDS d-values matched up well with the calculated d-values that were produced by the refinement programs. Small errors are noted. For instance, fluorapatite showed the greatest error between observed and calculated 2-theta was .041°. This corresponds to a very small distance on a powder film or X-ray strip chart. Other errors are the misindexing of d-values. Excluding germanite, this only occurred once, with hardystonite. On the other hand , though there are some JCPDS cards which have many errors. In the case of germanite there were many unindexed lines, as well as lines that were indexed incorrectly. Murdoch probably realized this but still recorded these lines because he saw them and as a scientist he couldn't ignore them. He left his data for others to study and possibly come up with answers to the discrepancies he encountered.

Least-Squares Refinement of Cell Parameters
Tetragonal and Hexagonal

Theory:

Consider the d-spacing formulas:

(eqn. 1) $\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2}$ (tetragonal), $\frac{1}{d^2} = \frac{4(h^2+hk+k^2)}{3a^2} + \frac{l^2}{c^2}$ (hexagonal).

Let $D = \frac{1}{d^2}$, $A = \frac{1}{a^2}$, $C = \frac{1}{c^2}$, $r = h^2+k^2$ or $\frac{4}{3}(h^2+hk+k^2)$, $s = l^2$. Then eqn. 1 becomes a linear equation in two variables, A and C:

(eqn. 2) $$D = rA + sC$$

The "normal" equations for least-squares solution of this linear equation are the following:

$$[\Sigma r_i^2]A + [\Sigma r_i s_i]C = \Sigma r_i D_i$$

$$[\Sigma r_i s_i]A + [\Sigma s_i^2]C = \Sigma s_i D_i$$

Solution of these two equations yields A and C, from which a and c can be obtained. Let the matrix $B_{ij} = \begin{bmatrix} \Sigma r^2 & \Sigma rs \\ \Sigma rs & \Sigma s^2 \end{bmatrix}$ and B_{ij}^{-1} its inverse so that $B \cdot B^{-1} = 1$.

The variance of the unknowns, A and C, is then given by the following:

$\sigma_A^2 = B_{11}^{-1}\left(\frac{f}{n-2}\right)$, $\sigma_C^2 = B_{22}^{-1}\left(\frac{f}{n-2}\right)$ where f is the sum of the squares of the "residuals", i.e., the differences between D_o (derived from the observed d's) and D_c (calculated using the values of A and C). From these relationships, it is possible to determine the errors of the cell parameters, σ_a and σ_c, using the equations $\sigma_a^2 = \left(\frac{\partial a}{\partial A}\right)^2 \sigma_A^2$ and $\sigma_c^2 = \left(\frac{\partial c}{\partial C}\right)\sigma_C^2$, so that $\sigma_a = \pm\frac{1}{2}\left[\frac{B_{11}^{-1} f}{A^3 (n-2)}\right]^{1/2}$ and $\sigma_c = \pm\frac{1}{2}\left[\frac{B_{22}^{-1} f}{C^3 (n-2)}\right]^{1/2}$.

Method (using attached worksheet):

1. Enter n values of hkl and d_o (observed).
2. Calculate n values of r, s, and D_o.
3. Calculate the five sums which are functions of r, s, and D_o and enter into matrix (1).
4. Solve for unknowns and inverse of matrix (using Gauss-Jordan method):
   a. Divide each term of 1st row of matrix (1) by B_{11} and place quotients in 1st row of matrix (2); divide each term of 2nd row of matrix (1) by B_{21} and place quotients in 2nd row of matrix (2)
   b. Rewrite 1st row of matrix (2) in 1st row of matrix (3); subtract terms of 1st row of matrix (2) from corresponding terms of 2nd row and place in 2nd row of matrix (3).
   c. Divide each term in 1st row of matrix (3) by the term now in B_{12} position of matrix (3) and place quotients in 1st row of matrix (4); divide each term in 2nd row of matrix (3) by term now in B_{22} position of matrix (3) and place quotients in 2nd row of matrix (4).
   d. Subtract terms of 2nd row of matrix (4) from corresponding terms of 1st row and place in 1st row of matrix (5); rewrite 2nd row of matrix (4) in 2nd row of matrix (5) and of matrix (6)
   e. Divide each term in 1st row of matrix (5) by term now in B_{11} position and place quotients in 1st row of matrix (6)
5. Calculate n values of D_c (calculated), Δ, and d_c.
6. Calculate f.
7. Calculate a, c, σ_a, and σ_c

— All work should be recorded to at least 5 significant figures; results of a, c, σ_a, and σ_c should be given to 3 figures to the right of the decimal point.

Worksheet for Least-Squares Refinement of Cell Parameters

Substance: Portlandite - $Ca(OH)_2$ System: Hexagonal

hkl	d_o	r	s	D_o	D_c	Δ	d_c
001	4.90	0	1	.041649	.041623	.000026	4.902
100	3.112	4/3	0	.10326	.10318	.00008	3.113
101	2.628	4/3	1	.14479	.14480	−.00001	2.628
002	2.447	0	4	.16701	.16649	.00052	2.451
102	1.927	4/3	4	.26930	.26967	−.00037	1.926
110	1.796	4	0	.31002	.30954	.00048	1.797
111	1.687	4	1	.35137	.35116	.00021	1.688
003	1.634	0	9	.37454	.37461	−.00007	1.634
200	1.557	16/3	0	.41250	.41272	−.00022	1.557
201	1.484	16/3	1	.45408	.45434	−.00026	1.484

$r = h^2 + k^2$ (tetragonal)
$= 4/3(h^2 + hk + k^2)$ (hexagonal)
$s = l^2$
$D_o = 1/d_o^2$

$D_c = rA + sC$
$\Delta = D_o - D_c$
$d_c = (1/D_c)^{1/2}$

$\Sigma r^2 =$ 94.222 $\Sigma rD_o =$ 7.9571
$\Sigma rs =$ 16.000 $\Sigma sD_o =$ 6.1080
$\Sigma s^2 =$ 117.00 $f = \Sigma\Delta^2 =$ 8.10×10^{-7}

B_{ij}

(1)

$\Sigma r^2 =$ 94.222	$\Sigma rs =$ 16	$\Sigma rD_o =$ 7.9571	1	0
$\Sigma rs =$ 16	$\Sigma s^2 =$ 117	$\Sigma sD_o =$ 6.1080	0	1

(2)

1	.16981	.084451	.010613	0
1	7.3125	.38175	0	.062500

(3)

1	.16981	.084451	.010613	0
0	7.1427	.29730	−.010613	.062500

(4)

5.8889	1	.49733	.062499	0
0	1	.041623	−.0014859	.0087502

(5)

5.8889	0	.45571	.063985	−.0087502
0	1	.041623	−.0014859	.0087502

(6)

1	0	$A =$ .077385	$B_{11}^{-1} =$ .010865	$B_{12}^{-1} =$ −.0014859
0	1	$C =$ .041623	$B_{21}^{-1} =$ −.0014859	$B_{22}^{-1} =$ .0087502

B_{ij}^{-1}

$a = (1/A)^{1/2}$, $\sigma_a = \pm\frac{1}{2}\left[\frac{B_{11}^{-1} f}{A^3 (n-2)}\right]^{1/2}$

$c = (1/C)^{1/2}$, $\sigma_c = \pm\frac{1}{2}\left[\frac{B_{22}^{-1} f}{C^3 (n-2)}\right]^{1/2}$

$a =$ 3.595 $\pm$ .001

$c =$ 4.902 $\pm$ .002

Checks: $B_{12}^{-1} = B_{21}^{-1}$ ✓

$B_{11} \cdot B_{11}^{-1} + B_{12} \cdot B_{21}^{-1} =$ .99995 ✓

$B_{21} \cdot B_{12}^{-1} + B_{22} \cdot B_{22}^{-1} = 1$ ✓

EMBOLITE 4-255

CUBIC

LAMBDA = 1.540562A; INPUT DATA: 14 VALUES OF D(OBS)

A = 5.6273A
+/- 0.0017

H	K	L	2-THETA	D(OBS)	(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	1	27.506	3.2400	3.2489	0.00052	0.077	1.00
2	0	0	31.819	2.8100	2.8137	0.00033	0.042	1.00
2	2	0	45.570	1.9890	1.9896	0.00014	0.013	1.00
3	1	1	53.955	1.6980	1.6967	-0.00053	-0.045	1.00
2	2	2	56.667	1.6230	1.6245	0.00068	0.056	1.00
4	0	0	66.333	1.4080	1.4068	-0.00084	-0.062	1.00
3	3	1	73.327	1.2900	1.2910	0.00093	0.066	1.00
4	2	0	75.513	1.2580	1.2583	0.00031	0.022	1.00
4	2	2	83.925	1.1520	1.1487	-0.00437	-0.299	1.00
4	4	0	101.739	0.9930	0.9948	0.00362	0.251	1.00
6	0	0	110.409	0.9380	0.9379	-0.00028	-0.020	1.00
4	4	2	110.409	0.9380	0.9379	-0.00028	-0.020	1.00
6	2	0	120.099	0.8890	0.8898	0.00215	0.169	1.00
6	2	2	130.264	0.8490	0.8484	-0.00213	-0.190	1.00

A2.1
Refinement output
for embolite

EMBOLITE 4-255 HKL GENERATOR

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	1	1	3.2482																				
2	1	0	2.8130																				
2	2	0	1.9891																				
3	1	1	1.6963																				
2	2	2	1.6241																				
4	0	0	1.4065																				
3	3	1	1.2907																				
4	2	0	1.2580																				
4	2	2	1.1484																				
5	1	1	1.0827																				
3	3	3	1.0827																				
4	4	0	0.9945																				
5	3	1	0.9510																				
6	0	0	0.9377																				
4	4	2	0.9377																				
6	2	0	0.8895																				
5	3	3	0.8580																				
6	2	2	0.8482																				

A2.2
hkl generator output
for embolite

12-453 HARDYSTONITE

TETRAGONAL

LAMBDA = 1.540562A; INPUT DATA: 35 VALUES OF D(OBS)

A3.1
Refinement output
for hardystonite

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF 1/D**2

A= 7.8187A +/- 0.0026 C= 5.0160A 0.0018

SSR (DEGREES 2-THETA): 1.5273D-01
RMS RESIDUALS (DEGREES 2-THETA): 6.6058D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
0	0	1	17.660	5.0180	5.0160	-0.00003	-0.007	1.00
1	0	1	21.034	4.2200	4.2219	0.00005	0.010	1.00
1	1	1	23.960	3.7110	3.7149	0.00015	0.026	1.00
2	1	0	25.479	3.4930	3.4966	0.00017	0.027	1.00
2	0	1	28.918	3.0850	3.0835	-0.00010	-0.015	1.00
2	1	1	31.159	2.8680	2.8685	0.00004	0.005	1.00
2	2	0	32.184	2.7790	2.7643	-0.00138	-0.176	1.00
0	0	2	35.802	2.5060	2.5080	0.00026	0.030	1.00
3	1	0	36.296	2.4730	2.4725	-0.00007	-0.008	1.00
2	2	1	37.120	2.4200	2.4210	0.00014	0.016	1.00
3	0	2	37.702	2.3840	2.3882	0.00061	0.068	1.00
3	0	1	38.887	2.3140	2.3127	-0.00021	-0.023	1.00
3	1	2	39.437	2.2830	2.2840	0.00017	0.018	1.00
3	1	1	40.624	2.2190	2.2177	-0.00024	-0.025	1.00
3	2	2	44.438	2.0370	2.0380	0.00023	0.022	1.00
4	2	1	45.521	1.9910	1.9905	-0.00013	-0.013	1.00
4	0	0	46.559	1.9490	1.9547	0.00152	0.143	1.00
4	1	0	47.941	1.8960	1.8963	0.00009	0.008	1.00
4	2	2	49.041	1.8560	1.8575	0.00045	0.041	1.00
3	1	2	51.878	1.7610	1.7607	-0.00010	-0.008	1.00
3	3	1	52.879	1.7300	1.7298	-0.00007	-0.006	1.00
4	2	2	56.027	1.6400	1.6404	0.00017	0.014	1.00
1	1	3	57.557	1.6000	1.6004	0.00021	0.017	1.00
2	0	3	60.153	1.5370	1.5373	0.00017	0.013	1.00
4	1	3	61.434	1.5080	1.5084	0.00025	0.019	1.00
1	1	1	63.298	1.4680	1.4664	-0.00102	-0.078	1.00
2	2	3	65.133	1.4310	1.4307	-0.00023	-0.017	1.00
0	0	3	66.386	1.4070	1.4073	0.00022	0.016	1.00
4	2	1	67.032	1.3950	1.3946	-0.00026	-0.019	1.00
1	1	3	67.581	1.3850	1.3850	0.00003	0.002	1.00
4	3	2	70.783	1.3300	1.3269	-0.00261	-0.188	1.00
5	1	2	72.350	1.3050	1.3082	0.00290	0.207	1.00
6	0	0	72.479	1.3030	1.3031	0.00010	0.007	1.00
4	1	3	75.796	1.2540	1.2541	0.00013	0.009	1.00
1	0	4	76.880	1.2390	1.2382	-0.00086	-0.060	1.00

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 7.8193A +/- 0.0027 C= 5.0154A 0.0019

SSR (DEGREES 2-THETA): 1.5213D-01
RMS RESIDUALS (DEGREES 2-THETA): 6.5928D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
0	0	1	17.660	5.0180	5.0154	-0.00004	-0.009	1.00
1	0	1	21.034	4.2200	4.2216	0.00004	0.008	1.00
1	1	1	23.960	3.7110	3.7148	0.00015	0.025	1.00
2	1	0	25.479	3.4930	3.4969	0.00018	0.029	1.00
2	0	1	28.918	3.0850	3.0835	-0.00011	-0.015	1.00
2	1	1	31.159	2.8680	2.8685	0.00004	0.005	1.00
2	2	0	32.184	2.7790	2.7645	-0.00136	-0.173	1.00
0	0	2	35.802	2.5060	2.5077	0.00021	0.025	1.00
3	1	0	36.296	2.4730	2.4727	-0.00004	-0.005	1.00
2	1	1	37.120	2.4200	2.4211	0.00015	0.017	1.00
3	0	2	37.702	2.3840	2.3879	0.00057	0.064	1.00
3	0	1	38.887	2.3140	2.3128	-0.00020	-0.022	1.00
3	1	2	39.437	2.2830	2.2838	0.00013	0.014	1.00
3	1	1	40.624	2.2190	2.2178	-0.00022	-0.023	1.00
3	2	2	44.438	2.0370	2.0379	0.00020	0.020	1.00
4	2	1	45.521	1.9910	1.9905	-0.00011	-0.011	1.00
4	0	0	46.559	1.9490	1.9548	0.00156	0.147	1.00
4	1	0	47.941	1.8960	1.8964	0.00013	0.012	1.00
4	2	2	49.041	1.8560	1.8574	0.00043	0.039	1.00
3	1	2	51.878	1.7610	1.7607	-0.00011	-0.010	1.00
3	3	1	52.879	1.7300	1.7299	-0.00003	-0.003	1.00
4	2	2	56.027	1.6400	1.6403	0.00016	0.013	1.00
1	1	3	57.557	1.6000	1.6002	0.00012	0.009	1.00
2	0	3	60.153	1.5370	1.5372	0.00009	0.007	1.00
4	1	3	61.434	1.5080	1.5083	0.00017	0.013	1.00
1	1	1	63.298	1.4680	1.4665	-0.00097	-0.074	1.00
2	2	3	65.133	1.4310	1.4306	-0.00030	-0.023	1.00
0	0	3	66.386	1.4070	1.4072	0.00014	0.011	1.00
4	2	1	67.032	1.3950	1.3947	-0.00020	-0.015	1.00
1	1	3	67.581	1.3850	1.3849	-0.00004	-0.003	1.00
4	3	2	70.783	1.3300	1.3270	-0.00259	-0.186	1.00
5	1	2	72.350	1.3050	1.3083	0.00292	0.209	1.00
6	0	0	72.479	1.3030	1.3032	0.00019	0.013	1.00
4	1	3	75.796	1.2540	1.2541	0.00008	0.006	1.00
1	0	4	76.880	1.2390	1.2380	-0.00102	-0.071	1.00

HARDYSTONITE 12-453 HKL GENERATOR

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	0	0	5.5317	3	1	3	1.3847								
0	1	1	5.0130	4	3	0	1.3829								
2	0	0	4.2208	5	4	1	1.3416								
1	1	0	4.0215	4	4	2	1.3331								
0	1	1	3.9146	5	5	0	1.3272								
2	0	1	3.7146	4	5	1	1.3272								
1	1	1	3.4986	5	4	2	1.3235								
2	0	0	3.0838	3	5	2	1.3085								
2	0	1	2.8690	5	5	1	1.3038								
2	1	0	2.7658	6	5	0	1.2966								
2	1	1	2.5065	5	5	2	1.2861								
2	2	0	2.4738	6	4	2	1.2704								
2	2	1	2.4217	6	5	1	1.2615								
2	0	2	2.3870	5	5	3	1.2565								
2	2	0	2.3134	6	5	2	1.2540								
2	2	1	2.2831	6	4	3	1.2532								
2	2	2	2.2184	6	6	0	1.2457								
2	1	2	2.1697	6	5	3	1.2382								
2	2	2	2.1104	7	4	1	1.2375								
2	0	3	2.0375	6	6	2	1.2369								
2	1	3	1.9912	7	5	0	1.2223								
1	1	0	1.9557	6	6	3	1.2217								
1	1	1	1.8974	7	5	2	1.2163								
1	1	2	1.8573	7	4	3	1.2083								
1	1	3	1.8439	6	6	4	1.2005								
1	1	4	1.8220												
1	1	5	1.8071												
1	1	6	1.7745												
1	1	7	1.7607												
1	1	8	1.7493												
1	1	9	1.7305												
1	1	10	1.6710												
1	1	11	1.6516												
1	1	12	1.6405												
1	1	13	1.6341												
1	1	14	1.5996												
1	1	15	1.5646												
1	1	16	1.5419												
1	1	17	1.5367												
1	1	18	1.5342												
1	1	19	1.5128												
1	1	20	1.5078												
1	1	21	1.4935												
1	1	22	1.4853												
1	1	23	1.4670												
1	1	24	1.4527												
1	1	25	1.4345												
1	1	26	1.4302												
1	1	27	1.4069												
1	1	28	1.3953												

'FLUORAPATITE 15-876

HEXAGONAL

LAMBDA = 1.540562A; INPUT DATA: 40 VALUES OF D(OBS)

A= 9.3712A C= 6.8849A
+/- 0.0006 0.0008

H	K	L	2-THETA	D(OBS)	(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	0	0	10.887	8.1200	8.1157	-0.00002	-0.006	1.00
1	0	1	16.874	5.2500	5.2502	0.00000	0.001	1.00
1	1	0	18.930	4.6840	4.6856	0.00003	0.007	1.00
2	0	0	21.901	4.0550	4.0578	0.00009	0.016	1.00
1	1	1	22.949	3.8720	3.8736	0.00006	0.010	1.00
2	0	1	25.472	3.4940	3.4958	0.00009	0.014	1.00
0	0	2	25.863	3.4420	3.4424	0.00002	0.003	1.00
1	0	2	28.153	3.1670	3.1691	0.00013	0.019	1.00
2	1	0	29.091	3.0670	3.0674	0.00003	0.004	1.00
2	1	1	31.936	2.8060	2.8019	0.00018	0.023	1.00
1	1	2	32.267	2.7720	2.7742	0.00021	0.026	1.00
3	0	0	33.127	2.7020	2.7052	0.00033	0.041	1.00
2	0	2	34.141	2.6240	2.6251	0.00012	0.014	1.00
3	0	1	35.640	2.5170	2.5178	0.00011	0.012	1.00
2	1	2	39.329	2.2890	2.2902	0.00019	0.021	1.00
3	1	0	40.040	2.2500	2.2509	0.00016	0.017	1.00
2	2	1	40.643	2.2180	2.2179	-0.00002	-0.002	1.00
3	1	1	42.194	2.1400	2.1395	-0.00011	-0.011	1.00
3	0	2	42.443	2.1280	2.1270	-0.00020	-0.020	1.00
1	1	3	43.893	2.0610	2.0610	0.00001	0.001	1.00
4	0	0	44.645	2.0280	2.0289	0.00022	0.021	1.00
2	0	3	45.377	1.9970	1.9976	0.00015	0.015	1.00
2	2	2	46.865	1.9370	1.9368	-0.00005	-0.005	1.00
3	1	2	48.266	1.8840	1.8839	-0.00003	-0.002	1.00
3	2	0	48.873	1.8620	1.8619	-0.00004	-0.004	1.00
2	1	3	49.583	1.8370	1.8376	0.00019	0.017	1.00
3	2	1	50.764	1.7970	1.7973	0.00011	0.009	1.00
4	1	0	51.563	1.7710	1.7710	-0.00000	-0.000	1.00
4	0	2	52.292	1.7480	1.7479	-0.00003	-0.003	1.00
0	0	4	53.143	1.7220	1.7212	-0.00031	-0.026	1.00
1	0	4	54.440	1.6840	1.6838	-0.00010	-0.008	1.00
3	2	2	56.139	1.6370	1.6377	0.00031	0.025	1.00
3	1	3	57.283	1.6070	1.6070	-0.00001	-0.001	1.00
5	0	1	58.355	1.5800	1.5798	-0.00009	-0.007	1.00
3	3	0	59.094	1.5620	1.5619	-0.00007	-0.006	1.00
4	2	0	60.283	1.5340	1.5337	-0.00015	-0.012	1.00
3	3	1	60.720	1.5240	1.5232	-0.00047	-0.037	1.00
2	1	4	61.752	1.5010	1.5011	0.00003	0.003	1.00
4	2	1	61.935	1.4970	1.4970	0.00002	0.001	1.00
5	0	2	63.298	1.4680	1.4681	0.00008	0.006	1.00

A4.1
Refinement output
of fluorapatite

FLUORAPATITE 15-876 HKL GENERATOR

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	0	0	8.1289	5	0	0	1.6258												
1	0	1	5.2534	1	1	4	1.6158												
1	1	0	4.6932	3	1	3	1.6082												
2	0	0	4.0644	1	3	3	1.6082												
1	0	1	3.8778	2	0	4	1.5848												
2	0	1	3.4999	5	0	1	1.5822												
0	0	2	3.4420	4	1	2	1.5768												
2	0	2	3.1696	1	4	2	1.5768												
2	1	0	3.0724	3	3	0	1.5644												
1	2	0	3.0724	4	2	0	1.5362												
2	1	1	2.8057	2	4	0	1.5362												
1	2	1	2.8057	3	3	1	1.5255												
1	1	2	2.7756	4	0	3	1.5214												
3	0	0	2.7096	2	1	4	1.5015												
3	0	2	2.6267	1	2	4	1.5015												
3	0	1	2.5213	4	2	1	1.4993												
2	2	0	2.3466	2	4	1	1.4993												
2	1	2	2.2921	5	0	2	1.4700												
1	2	2	2.2921	5	1	0	1.4600												
3	1	0	2.2545	1	5	0	1.4600												
1	3	0	2.2545	3	0	4	1.4528												
2	2	1	2.2211	3	3	3	1.4472												
1	3	1	2.2084	2	3	3	1.4472												
3	1	1	2.1426	5	1	1	1.4282												
1	3	1	2.1426	1	5	1	1.4282												
3	1	2	2.1291	3	3	2	1.4242												
1	4	0	2.0615	4	1	3	1.4034												
4	0	0	2.0322	1	4	3	1.4034												
2	0	3	1.9982	4	2	2	1.4028												
4	0	2	1.9491	2	4	2	1.4028												
2	2	2	1.9389																
3	1	2	1.8860																
1	3	2	1.8860																
3	2	0	1.8649																
2	3	0	1.8649																
2	1	3	1.8385																
1	3	2	1.8385																
4	0	1	1.8000																
3	2	1	1.8000																
4	1	0	1.7739																
1	4	0	1.7739																
4	0	3	1.7511																
3	2	2	1.7500																
4	0	0	1.7210																
2	4	1	1.7178																
4	0	1	1.7178																
1	4	2	1.6837																
4	1	3	1.6406																
2	3	2	1.6397																
3	2	2	1.6397																

A4.2
hkl generator output
of fluorapatite

FRANKLINITE 10-467

CUBIC

LAMBDA = 1.540562A; INPUT DATA: 17 VALUES OF D(OBS)

Refinement output of franklinite

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF 1/D**2

A= 8.4768A
+/- 0.0017

SSR (DEGREES 2-THETA): 9.7681D-02
RMS RESIDUALS (DEGREES 2-THETA): 7.5802D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	1	18.164	4.8800	4.8941	0.00024	0.053	1.00
2	2	0	29.858	2.9900	2.9970	0.00052	0.071	1.00
3	1	1	35.164	2.5500	2.5558	0.00070	0.083	1.00
2	2	2	36.805	2.4400	2.4470	0.00096	0.109	1.00
4	0	0	42.611	2.1200	2.1192	-0.00017	-0.017	1.00
4	2	2	52.879	1.7300	1.7303	0.00012	0.010	1.00
5	1	1	56.327	1.6320	1.6314	-0.00030	-0.024	1.00
4	4	0	61.843	1.4990	1.4985	-0.00030	-0.023	1.00
6	2	0	70.176	1.3400	1.3403	0.00024	0.018	1.00
5	3	3	73.130	1.2930	1.2927	-0.00028	-0.020	1.00
6	2	2	74.063	1.2790	1.2779	-0.00104	-0.073	1.00
4	4	4	77.999	1.2240	1.2235	-0.00053	-0.037	1.00
6	4	2	85.665	1.1330	1.1328	-0.00034	-0.023	1.00
7	3	1	88.488	1.1040	1.1036	-0.00063	-0.043	1.00
8	0	0	93.103	1.0610	1.0596	-0.00236	-0.161	1.00
6	6	0	101.036	0.9980	0.9990	0.00200	0.138	1.00
7	5	1	103.925	0.9780	0.9788	0.00173	0.121	1.00

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 8.4763A
+/- 0.0019

SSR (DEGREES 2-THETA): 9.7273D-02
RMS RESIDUALS (DEGREES 2-THETA): 7.5643D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	1	18.164	4.8800	4.8938	0.00024	0.052	1.00
2	2	0	29.858	2.9900	2.9968	0.00051	0.069	1.00
3	1	1	35.164	2.5500	2.5557	0.00068	0.081	1.00
2	2	2	36.805	2.4400	2.4469	0.00094	0.107	1.00
4	0	0	42.611	2.1200	2.1191	-0.00020	-0.020	1.00
4	2	2	52.879	1.7300	1.7302	0.00008	0.007	1.00
5	1	1	56.327	1.6320	1.6313	-0.00034	-0.028	1.00
4	4	0	61.843	1.4990	1.4984	-0.00035	-0.027	1.00
6	2	0	70.176	1.3400	1.3402	0.00018	0.013	1.00
5	3	3	73.130	1.2930	1.2926	-0.00035	-0.025	1.00
6	2	2	74.063	1.2790	1.2778	-0.00110	-0.078	1.00
4	4	4	77.999	1.2240	1.2234	-0.00061	-0.042	1.00
6	4	2	85.665	1.1330	1.1327	-0.00043	-0.029	1.00
7	3	1	88.488	1.1040	1.1035	-0.00072	-0.049	1.00
8	0	0	93.103	1.0610	1.0595	-0.00246	-0.168	1.00
6	6	0	101.036	0.9980	0.9989	0.00188	0.131	1.00
7	5	1	103.925	0.9780	0.9788	0.00161	0.113	1.00

VS LOADER

OPTIONS USED - PRINT,NOMAP,NOLET,CALL,RES,NOTERM,SIZE=163840,NAME=**GO

TOTAL LENGTH BBBC
ENTRY ADDRESS BC01C

FRANKLINITE 10-467 HKL GENERATOR PAGE= 1

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	1	1	4.8925																				
2	2	0	2.9960																				
3	1	2	2.5550																				
4	2	0	2.4462																				
3	2	1	2.1185																				
4	2	2	1.9441																				
5	1	3	1.7297																				
3	3	0	1.6308																				
4	3	1	1.4980																				
5	3	2	1.4324																				
4	4	0	1.4123																				
6	2	0	1.3399																				
5	3	2	1.2923																				
6	2	4	1.2775																				
4	2	4	1.2231																				
7	1	1	1.1866																				
5	1	5	1.1866																				
6	4	2	1.1324																				
7	3	1	1.1032																				
5	3	5	1.1032																				
8	0	3	1.0592																				
7	0	3	1.0353																				
6	4	4	1.0276																				
6	6	0	0.9987																				
8	2	2	0.9987																				
7	5	1	0.9785																				
5	5	5	0.9785																				
6	6	2	0.9720																				

A5.2
hkl generator output
of franklinite

A6.1
hkl generator output
of germanite

GERMANITE 10-469 HKL GENERATOR

PAGE= 1

H	K	L	D
1	2	2	7.4847
2	2	0	5.2925
1	3	1	4.4404
2	2	2	4.2473
3	3	0	3.5567
2	3	2	3.3738
3	3	2	2.9699
4	4	0	2.6462
3	4	2	2.5121
4	4	2	2.3738
5	5	1	2.2922
4	5	2	2.1784
5	5	2	2.0778
6	6	0	1.8690
5	6	2	1.8230
6	6	2	1.7736
7	7	1	1.7407
6	7	2	1.6953
7	7	2	1.6642
8	8	0	1.5597
7	8	2	1.5331
8	8	2	1.4969
9	9	1	1.4679
8	9	2	1.4540
9	9	2	1.4404

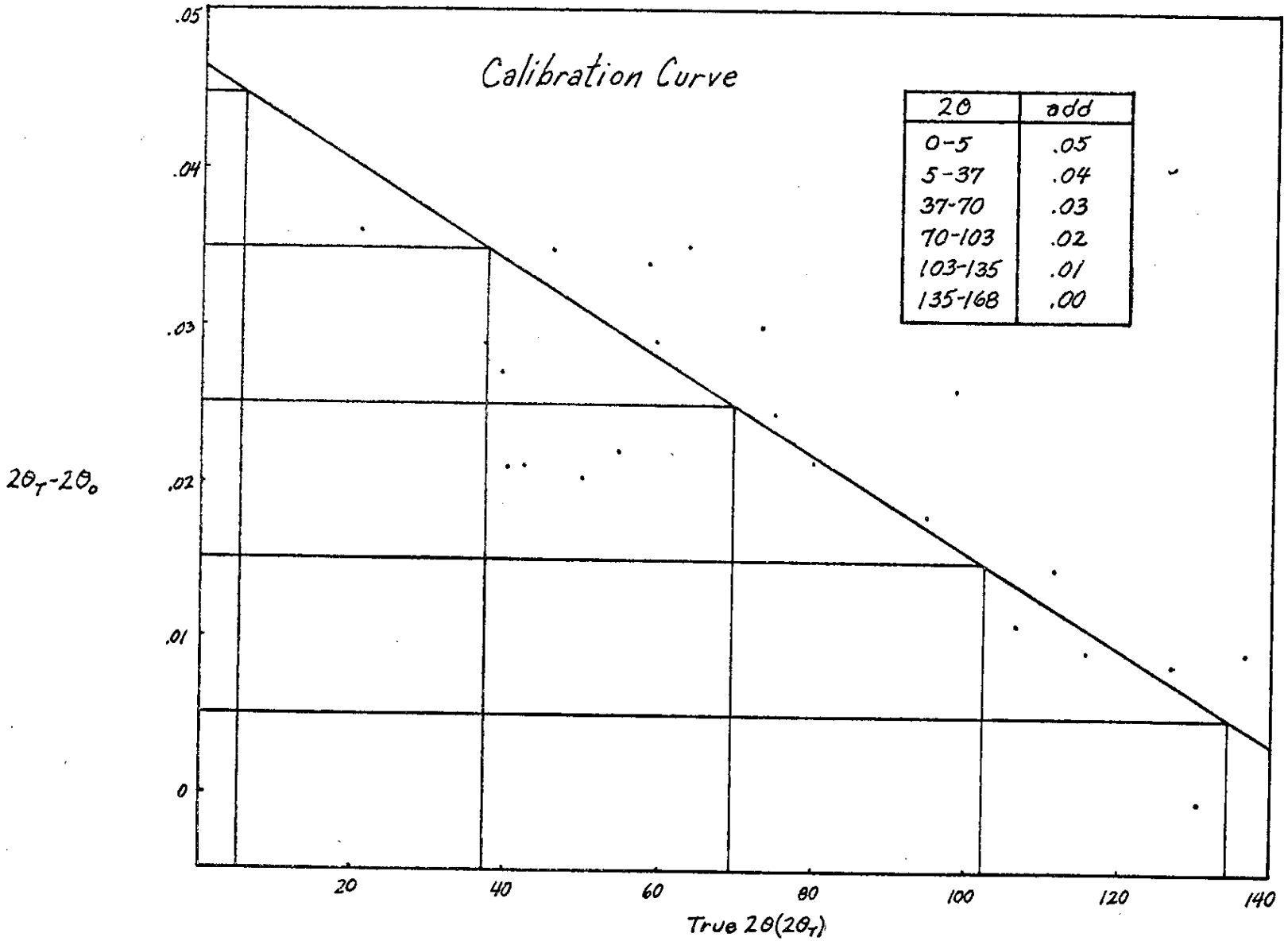
H	K	L	D
6	6	2	1.4404
7	7	0	1.4145
6	7	1	1.3889
7	7	2	1.3755
8	8	1	1.3644
7	8	2	1.3511
8	8	2	1.3384
9	9	0	1.3262
8	9	1	1.3144
9	9	1	1.3029
10	10	0	1.2918
9	10	1	1.2810
10	10	1	1.2705
11	11	0	1.2603
10	11	1	1.2503
11	11	1	1.2407
12	12	0	1.2313
11	12	1	1.2222
12	12	1	1.2133
13	13	0	1.2047
12	13	1	1.1963
13	13	1	1.1881
14	14	0	1.1802
13	14	1	1.1725
14	14	1	1.1650
15	15	0	1.1577
14	15	1	1.1506
15	15	1	1.1437

H	K	L	D
8	9	2	1.1370
9	9	2	1.1305
10	10	2	1.1241
11	11	2	1.1180
12	12	2	1.1120
13	13	2	1.1062
14	14	2	1.1006
15	15	2	1.0951
16	16	0	1.0898
15	16	1	1.0847
16	16	1	1.0797
17	17	0	1.0749
16	17	1	1.0702
17	17	1	1.0656
18	18	0	1.0612
17	18	1	1.0569
18	18	1	1.0527
19	19	0	1.0487
18	19	1	1.0447
19	19	1	1.0409
20	20	0	1.0372
19	20	1	1.0336
20	20	1	1.0301

H	K	L	D
10	11	2	1.0267
11	11	2	1.0234
12	12	2	1.0202
13	13	2	1.0171
14	14	2	1.0141
15	15	2	1.0112
16	16	2	1.0084
17	17	2	1.0057
18	18	2	1.0031
19	19	2	1.0006
20	20	2	.9982
21	21	0	.9958
20	21	1	.9936
21	21	1	.9914
22	22	0	.9893
21	22	1	.9873
22	22	1	.9853
23	23	0	.9834
22	23	1	.9816
23	23	1	.9798
24	24	0	.9781
23	24	1	.9764
24	24	1	.9748

H	K	L	D
12	13	2	.9732
13	13	2	.9717
14	14	2	.9702
15	15	2	.9688
16	16	2	.9674
17	17	2	.9661
18	18	2	.9648
19	19	2	.9636
20	20	2	.9624
21	21	2	.9613
22	22	2	.9602
23	23	2	.9591
24	24	2	.9581
25	25	0	.9571
24	25	1	.9562
25	25	1	.9552
26	26	0	.9543
25	26	1	.9535
26	26	1	.9526
27	27	0	.9518
26	27	1	.9510
27	27	1	.9503

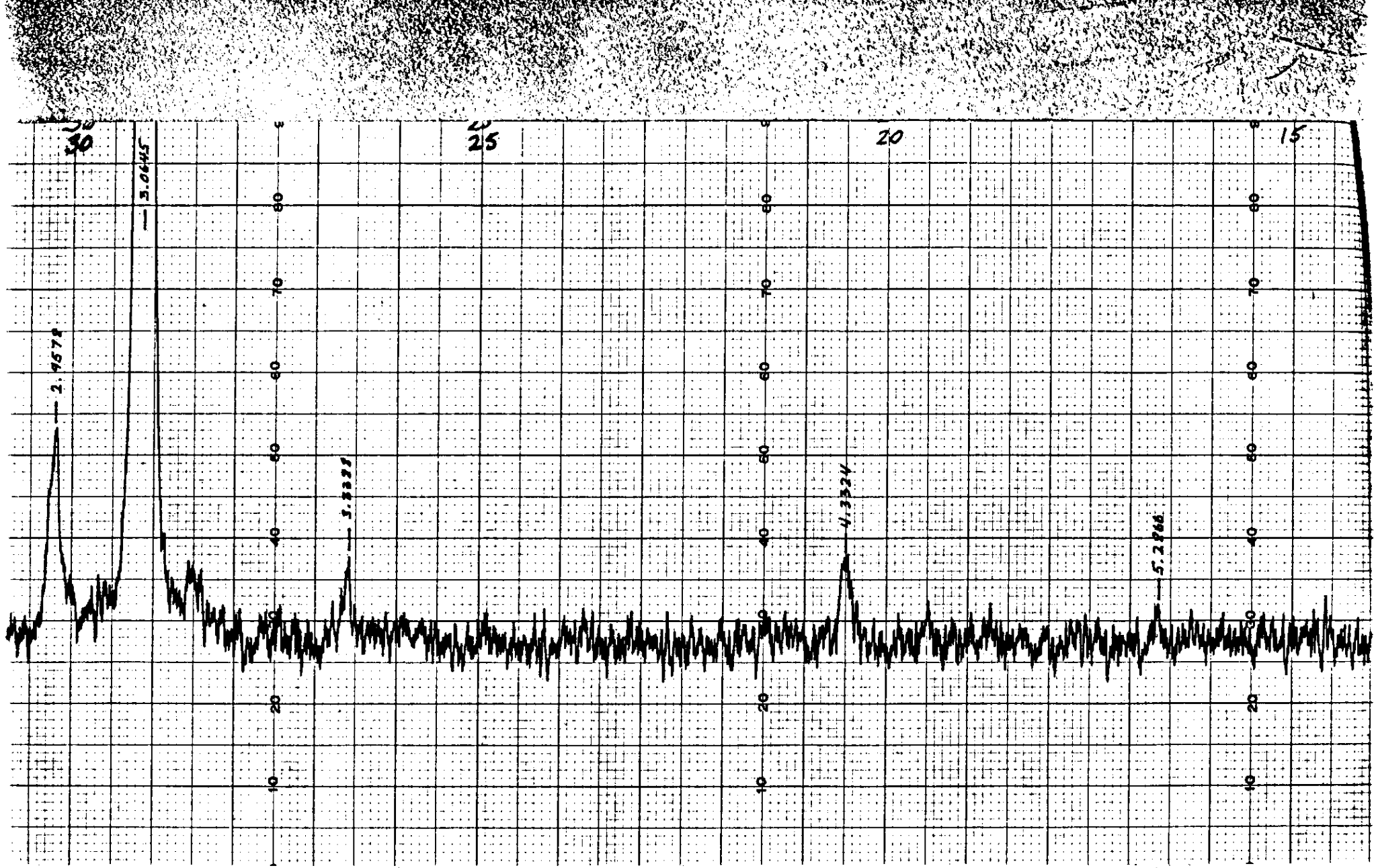
H	K	L	D
14	15	2	.9495
15	15	2	.9488
16	16	2	.9481
17	17	2	.9474
18	18	2	.9468
19	19	2	.9461
20	20	2	.9455
21	21	2	.9449
22	22	2	.9443
23	23	2	.9438
24	24	2	.9432
25	25	2	.9427
26	26	2	.9422
27	27	2	.9417
28	28	0	.9412
27	28	1	.9407
28	28	1	.9403
29	29	0	.9398
28	29	1	.9394
29	29	1	.9390
30	30	0	.9386
29	30	1	.9382
30	30	1	.9378



Calibration curve for adjusting 2θ-values of germanite with those of quartz. ($2\theta_T$ - accurate value of 2θ from Frondel (1962, pp. 26-33); $2\theta_o$ - value of 2θ read from X-ray strip chart.)

A6.2
Calibration curve for germanite

A6.3
X-ray strip chart
of germanite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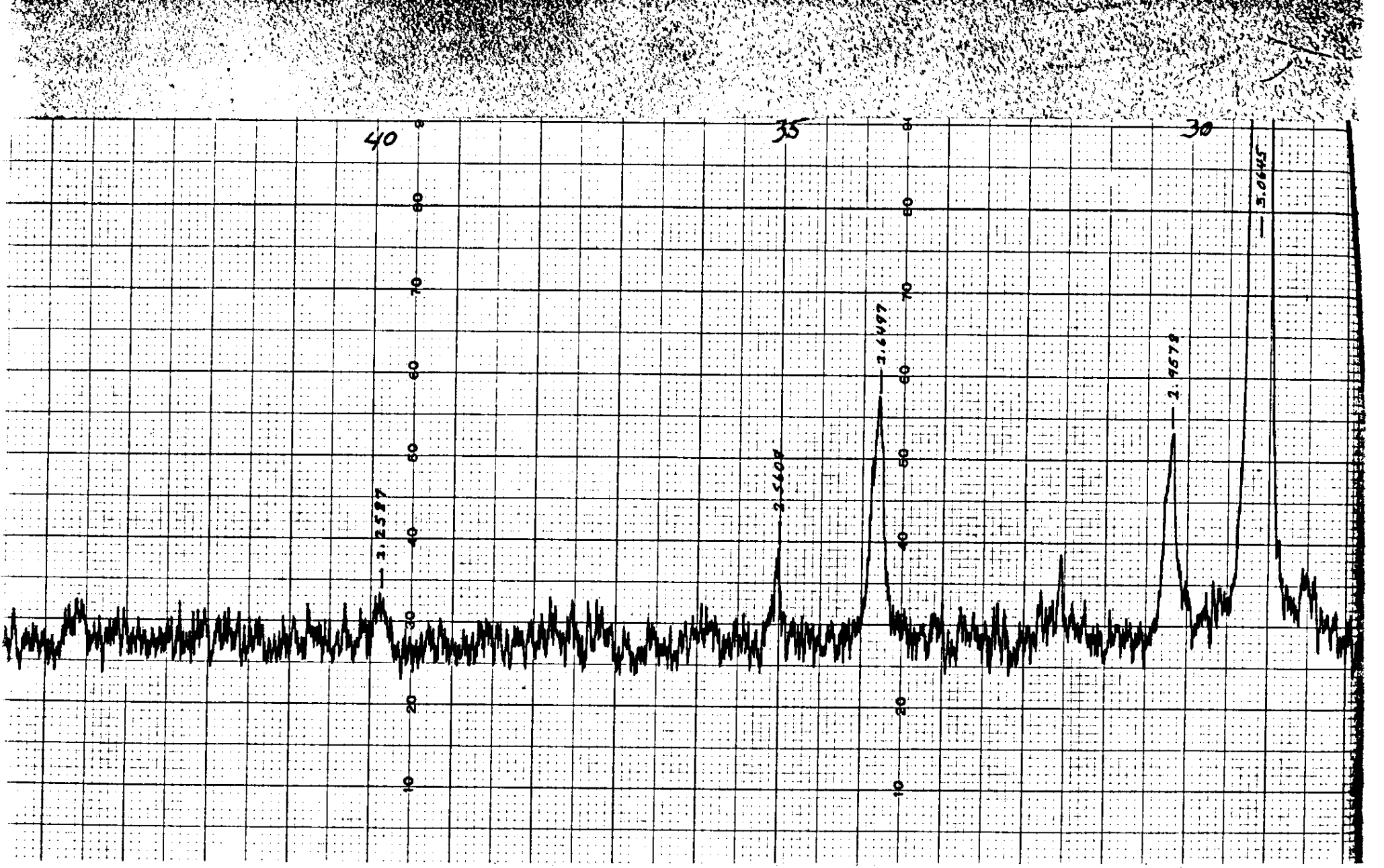
5.2988
2-theta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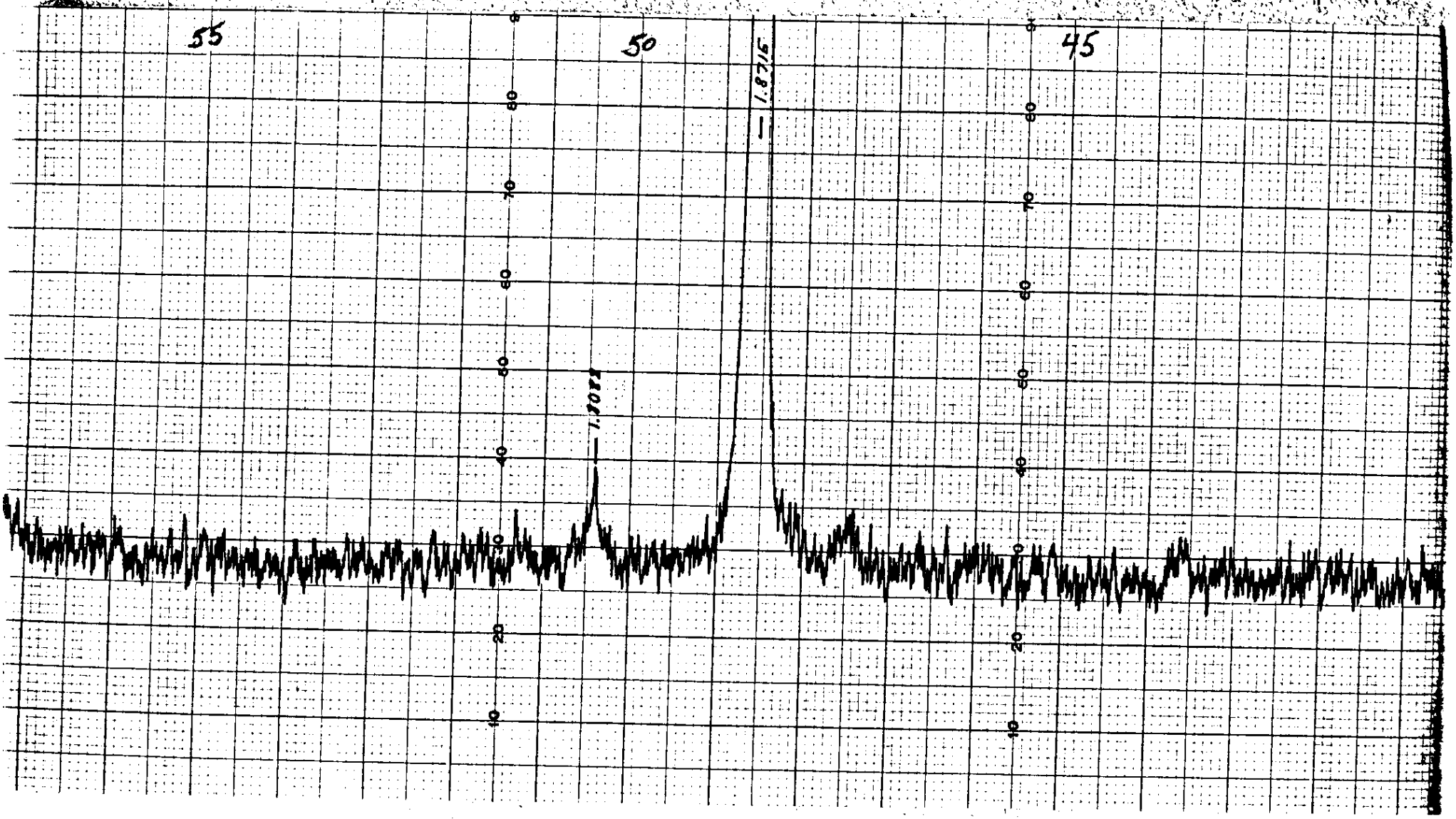
2-theta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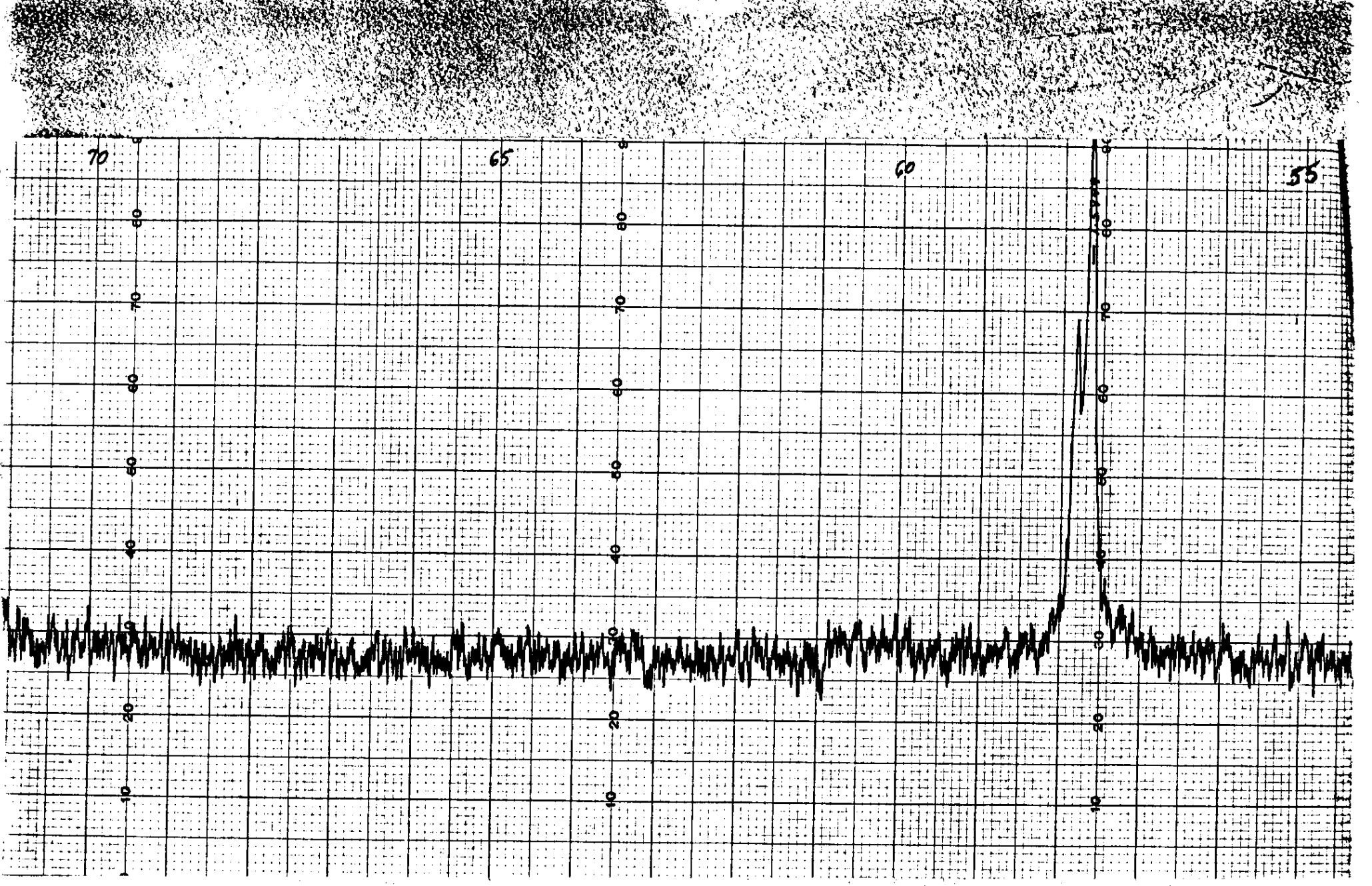
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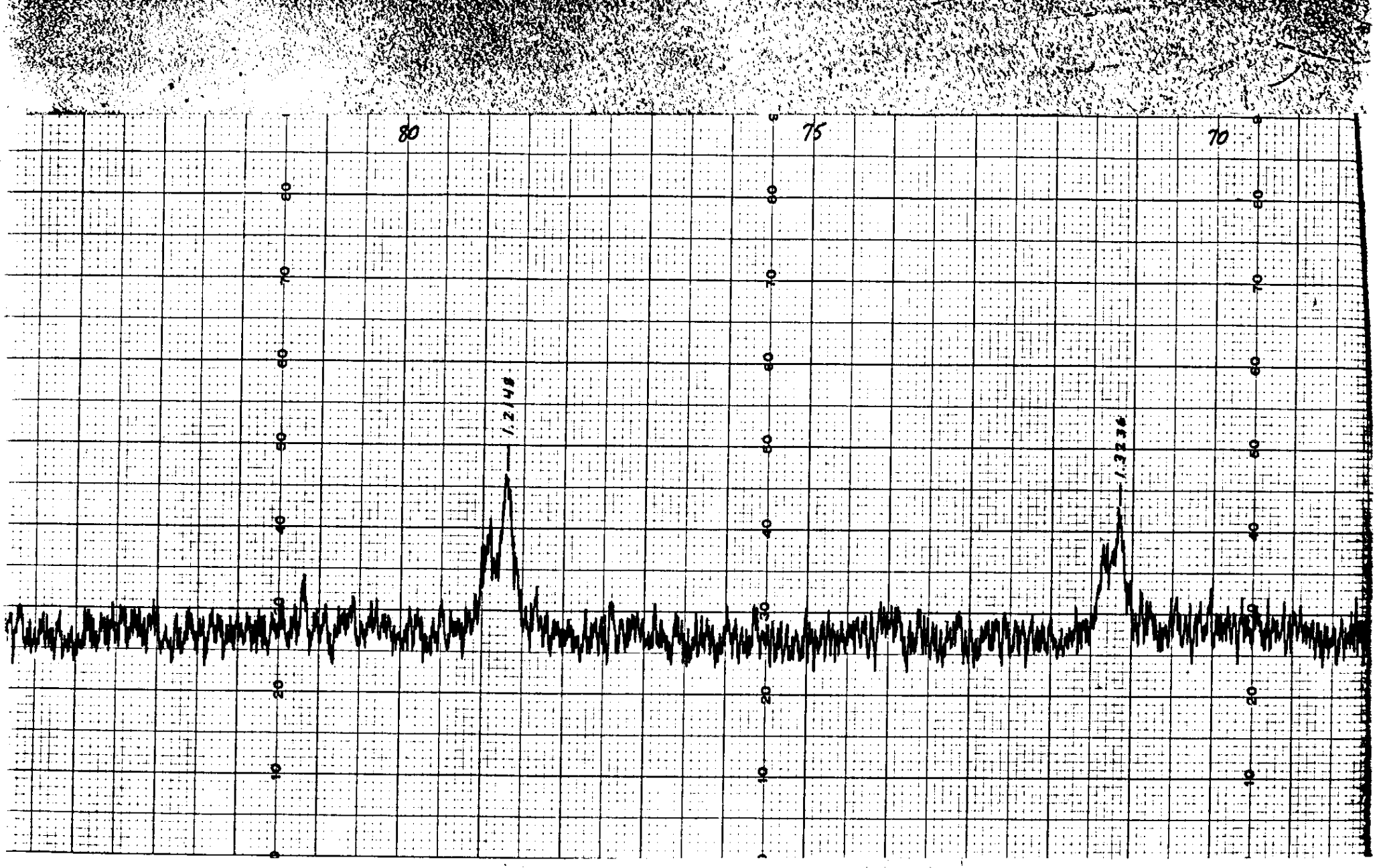
65
60
55
2-theta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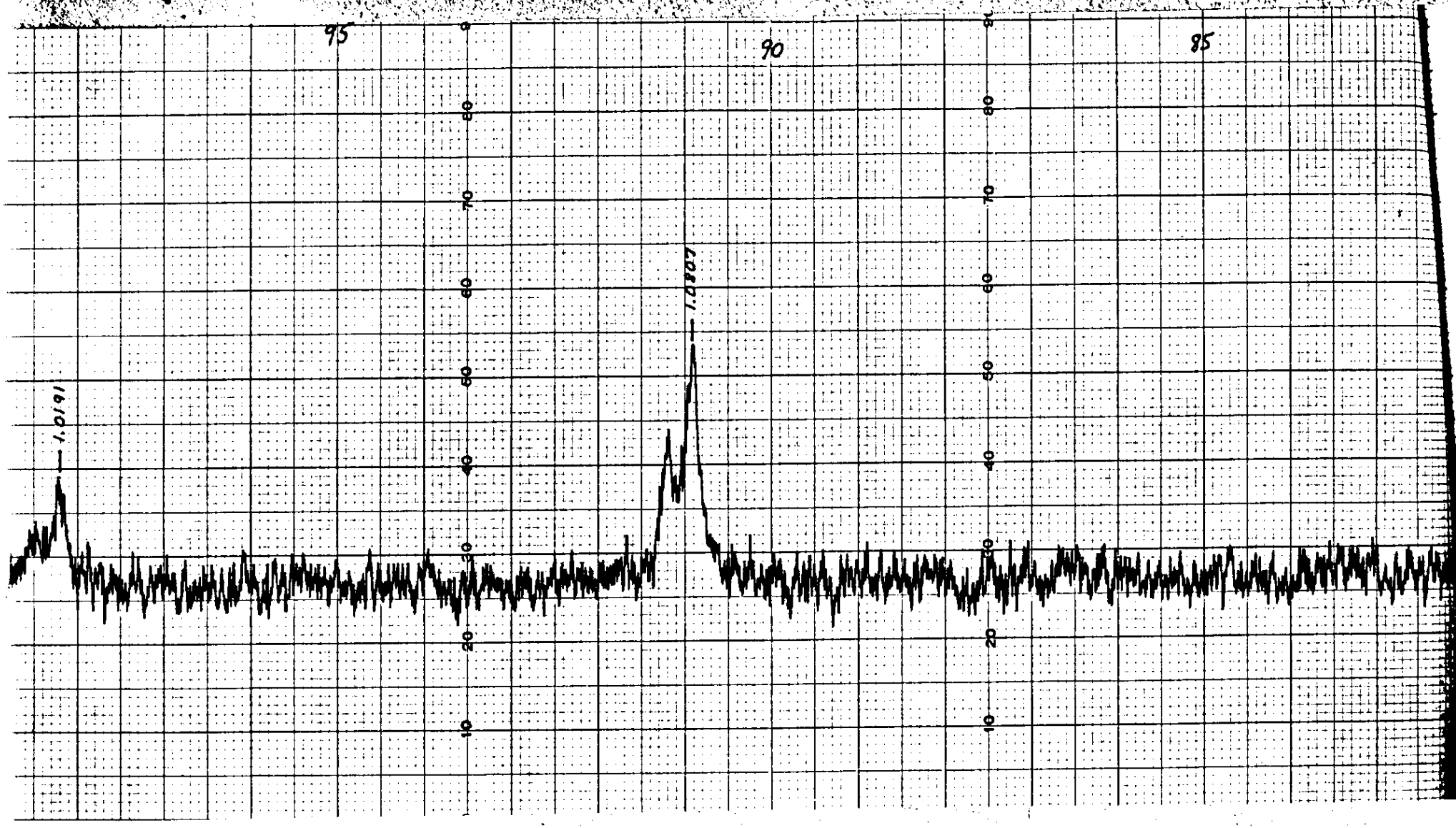
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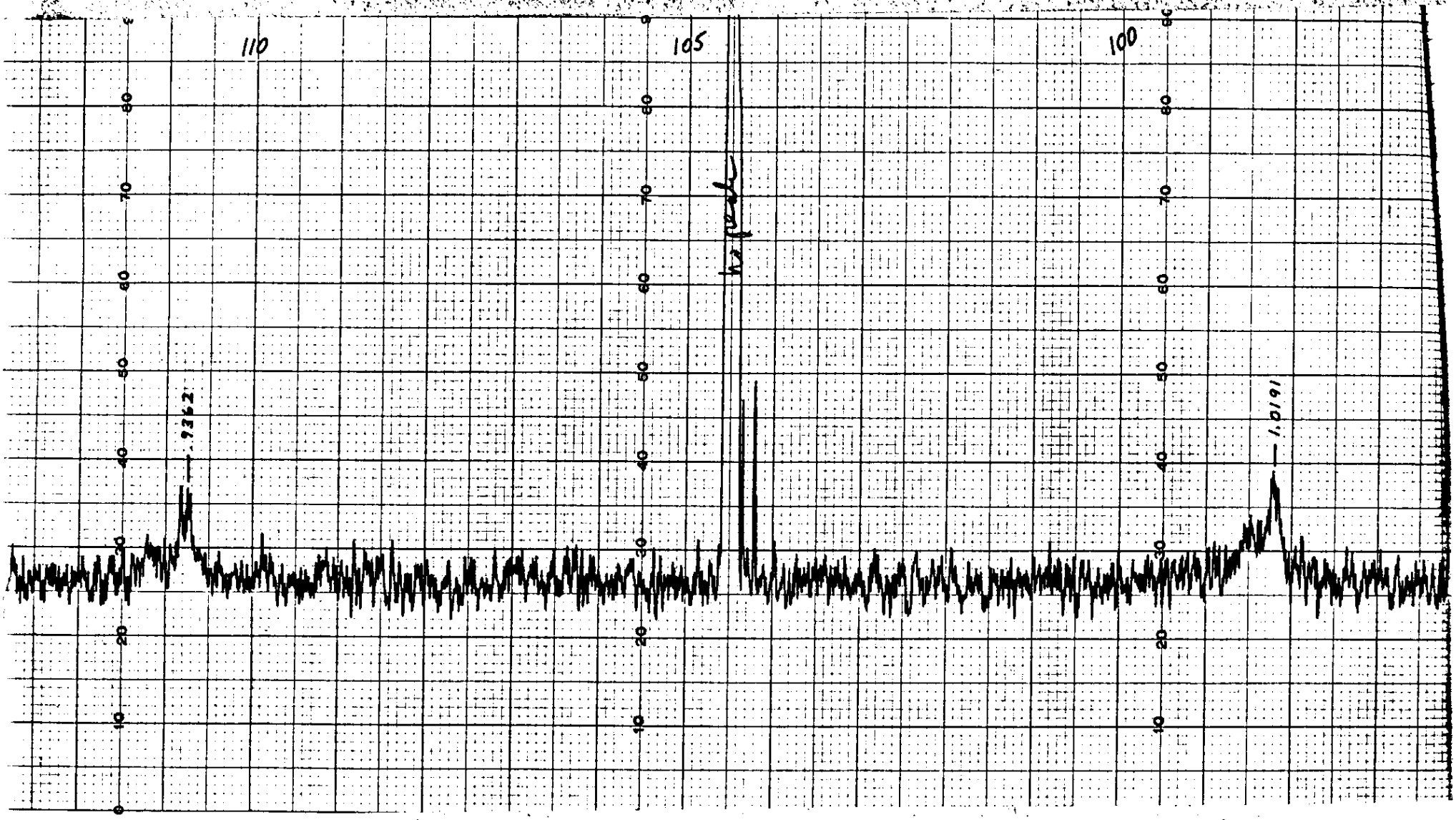
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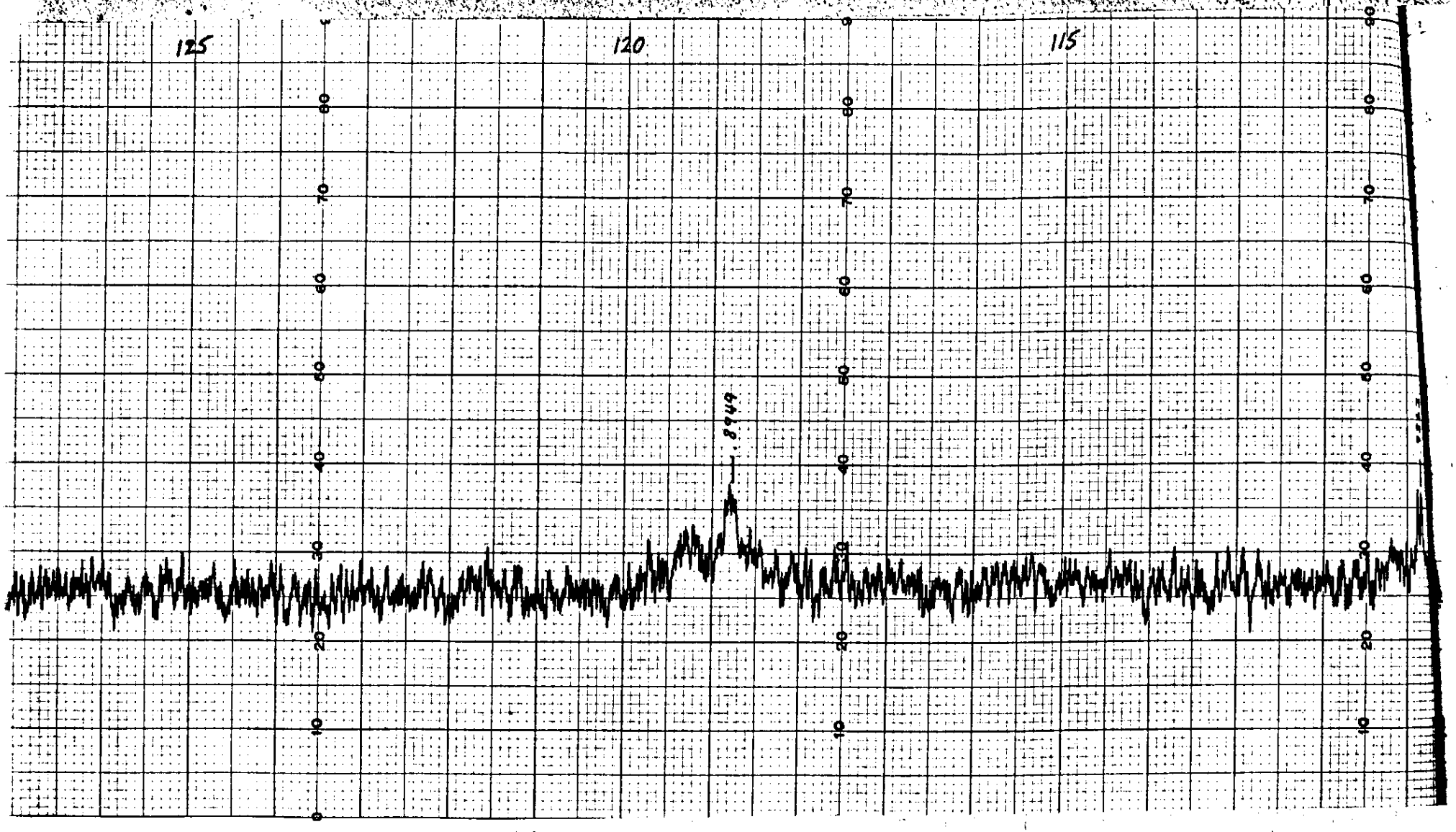
2-theta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115
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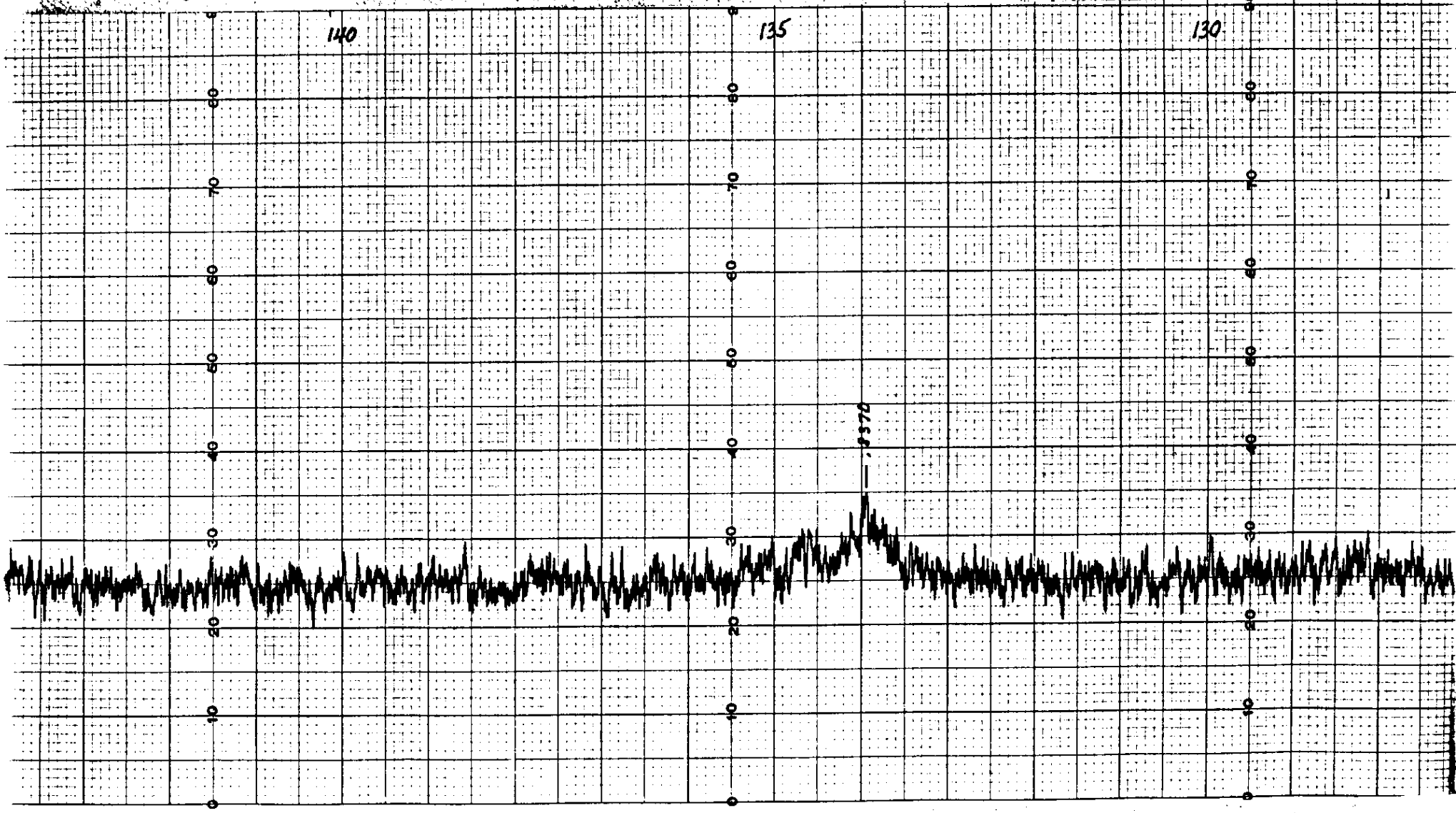

2-theta

GERMANITE 10-469

CUBIC

LAMBDA = 1.540562A; INPUT DATA: 20 VALUES OF 2-THETA

A6.4
Refinement output
of germanite

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF 1/D**2

A= 10.5732A
+/- 0.0063

SSR (DEGREES 2-THETA): 2.3409D+00
RMS RESIDUALS (DEGREES 2-THETA): 3.4212D-01

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
2	0	0	16.700	5.3042	5.2866	-0.00024	-0.056	1.00
2	1	1	20.540	4.3205	4.3165	-0.00010	-0.019	1.00
3	1	0	26.640	3.3434	3.3435	0.00001	0.001	1.00
2	2	2	29.180	3.0579	3.0522	-0.00040	-0.055	1.00
3	2	0	30.230	2.9540	2.9325	-0.00169	-0.227	1.00
4	0	0	33.840	2.6467	2.6433	-0.00037	-0.045	1.00
4	1	0	35.060	2.5573	2.5644	0.00084	0.099	1.00
3	3	2	39.910	2.2570	2.2542	-0.00049	-0.052	1.00
4	3	1	43.620	2.0733	2.0736	0.00007	0.007	1.00
4	4	0	48.640	1.8704	1.8691	-0.00039	-0.035	1.00
4	3	3	50.440	1.8078	1.8133	0.00186	0.164	1.00
6	2	2	57.690	1.5966	1.5940	-0.00131	-0.105	1.00
8	0	0	71.200	1.3232	1.3216	-0.00137	-0.098	1.00
6	6	2	78.900	1.2123	1.2128	0.00063	0.044	1.00
8	4	4	90.940	1.0805	1.0791	-0.00221	-0.150	1.00
6	6	6	98.220	1.0189	1.0174	-0.00290	-0.199	1.00
8	8	0	110.730	0.9362	0.9345	-0.00400	-0.291	1.00
10	6	2	118.800	0.8949	0.8936	-0.00365	-0.284	1.00
10	7	3	133.960	0.8369	0.8412	0.01433	1.338	1.00
10	6	6	145.220	0.8072	0.8062	-0.00372	-0.446	1.00

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 10.5725A
+/- 0.0064

SSR (DEGREES 2-THETA): 2.3394D+00
RMS RESIDUALS (DEGREES 2-THETA): 3.4201D-01

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
2	0	0	16.700	5.3042	5.2862	-0.00024	-0.057	1.00
2	1	1	20.540	4.3205	4.3162	-0.00011	-0.020	1.00
3	1	0	26.640	3.3434	3.3433	-0.00000	-0.001	1.00
2	2	2	29.180	3.0579	3.0520	-0.00041	-0.057	1.00
3	2	0	30.230	2.9540	2.9323	-0.00171	-0.230	1.00
4	0	0	33.840	2.6467	2.6431	-0.00036	-0.047	1.00
4	1	0	35.060	2.5573	2.5642	0.00082	0.097	1.00
3	3	2	39.910	2.2570	2.2541	-0.00052	-0.055	1.00
4	3	1	43.620	2.0733	2.0734	0.00004	0.004	1.00
4	4	0	48.640	1.8704	1.8690	-0.00043	-0.039	1.00
4	3	3	50.440	1.8078	1.8132	0.00182	0.161	1.00
6	2	2	57.690	1.5966	1.5939	-0.00136	-0.110	1.00
8	0	0	71.200	1.3232	1.3216	-0.00144	-0.104	1.00
6	6	2	78.900	1.2123	1.2127	0.00054	0.037	1.00
8	4	4	90.940	1.0805	1.0790	-0.00233	-0.158	1.00
6	6	6	98.220	1.0189	1.0173	-0.00303	-0.208	1.00
8	8	0	110.730	0.9362	0.9345	-0.00415	-0.302	1.00
10	6	2	118.800	0.8949	0.8935	-0.00382	-0.297	1.00
10	7	3	133.960	0.8369	0.8411	0.01414	1.321	1.00
10	6	6	145.220	0.8072	0.8061	-0.00393	-0.471	1.00

References

Cullity, B. D., 1956, Elements of X-ray Diffraction, Reading, Massachusetts, Addison-Wesley.

Frondel, C., 1962, The System of Mineralogy, 7 ed., v. 3: New York, Wiley

Murdoch, J., 1953, X-ray Investigation of Colusite, Germanite, and Renierite: American Mineralogist, v. 38, pp. 794-801.

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